IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: **Bisaria et al**
Serial No.: **09/479,712**
Filed: **January 7, 2000**

Attorney Docket No.: **CL1365USNA**
Group Art Unit: **1774**
Examiner: **Gray, J.**

For: **INJECTION MOLDABLE CONDUCTIVE AROMATIC THERMOPLASTIC LIQUID CRYSTALLINE POLYMERIC COMPOSITIONS**

Exhibit A to the

DECLARATION
of
Yuqi Cai

Under 37 C.F.R. § 1.131

LABORATORY NOTEBOOK

PROPERTY OF

DU PONT CANADA INC

RESEARCH & DEVELOPMENT

KINGSTON ONTARIO

No 2557

TITLE Conductive Composites BOOK NO.

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RESEARCH & DEVELOPMENT, KINGSTON (RDK)

Subject: **Operating Instructions** for: HIGH TEMPERATURE NYLON (**Proj.73132**)

Issue Date: 12th Sept. 1998
Expiry Date: 12th Sept 1999
Author: Yuqi Cai
Area: R&BD
Title: Conductive Composites
Department: Research & Development
Signed:__________
Date Approved:__________

EXPERIMENT NO.: 2557-05, (continuation of 2442-51)

This document has been reviewed by: E. Nielsen
D. A. Harbourne DAH
Yuqi Cai Cai

1. GENERAL INFORMATION:

1.1 PURPOSE:

To prepare an injection moldable conductive polymer composite which will have a resistivity of 0.01 ohms-cm or lower.

1.2 TIMING:

This project began with OI 2442-51 on June 15th 1998. OI 2557-20 is continuation of 2442-51.

1.3 BACKGROUND / PRIOR or RELATED EXPERIMENTS:

Fuel cell applications are being held back by the current high cost of fuel cell components, such as the "bipolar plate". Recent technology has moved the production process for this component from machining of graphite slabs to compression molding of polymer/conductive filler composites. The development of an injection moldable polymer/conductive filler composition would further reduce the cost into the range desired by fuel cell manufacturers such as Ballard Systems.

It is believed that the right combination of conductive fillers with the right base resin and the right compounding process, possibly with processing additives, is capable of providing a functional injection moldable composition.

LCP (DuPont Zenite*) compositions filled with conductive fillers are potential candidates. The expected range of filler level is between 50 to 80%. The types of fillers include Conoco's Thermocarb CF300, various carbon fibers, and possibly various carbon blacks.

Ballard have been shown an injection molded disc comprising LCP filled with about 70% by weight of a

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Conoco powdered graphite called "Thermocarb" . This sample provided a resistivity of about 1 ohm-cm. Ballard's target is 0.01 ohm-cm. Another sample comprising 30% LCP and 55% Thermocarb and 15% Hexcel carbon fibres (0.25") was mixed up in the small Brabender mixing head (m/h). Hot water washed. Waring blender "fluffed" fibres were used along with a "double pass" in the mixing head to provide a composition which when compression molded at about 30,000psi showed a resistivity of 0.05 ohm-cm. Ballard were shown this sample and impressed enough to request a follow up injection molded sample with equal or better (0.01) resistivity.

This work is directly focused on providing a composition to meet Ballard's needs
Through optical microscope analysis (OI2442-51and 2442-65, Fig.1-13), it can be observed that the Panex fibre were subject to severe breakage during W&P compounding and the following injection molding. The fibre length was observed to be less than 200 micrometers. No conductive network has been formed. The resisitivity of our injection molded plaques using Panex or Hexcel carbon fibres can not meet our target, even though the gentlest mixing in Brabender or dry blending were tried to avoid severe fibre breakage.

In the following work, Nickel-coated Carbon Fibres (NCF) are to be used in order to get longer fibre length in injection molded samples. We chose NCF because
(1) Nickel has excellent corrosion resistance. It is important for fuel cell bipolar plates
(2) Nickel-coated carbon fibres have lighter density (in comparison to 100% nickel fibres or stainless steel fibres) and much better conductivity (in comparison to any carbon fibres)
(3) Nickel-coated carbon fibres has acceptable price.

The metal coating should help prevent from fibre breakage and help form conductive network and therefore improve conductivity of the composites. NCF could be supplied in the form of unsized roving or chopped prepregs. We will use NCF or together with Thermocarb concentrate to fill LCP through compounding in Brabender mixer, W&E twin screw extruder or just dry blending. The compounded materials will be used for injection molding of conductive plaques.

1.4 EXPERIMENTAL SCOPE

1.4.1 Design Control:

The conductivity is potentially affected by the following parameters:

- Base resin characteristics
- Filler Type and loading
- Compounding shear/heat history

so that we need to investigate each of these in the design.

Therefore the design of this experiment revolves around:
1) specific composition
2) specific process
within conventional compounding methods.

While principles exist on which to base experiments, no models are available with which to set composition or process. We know that the conductive particles must "touch" (form a conductive network) to create a conductive path through the polymer.

We expect to be able to meet the target by some combination of conductive powder and some conductive fibre in a low viscosity base resin having adequate stability for the fuel cell application.
We expect to be able to mix the composition sufficiently well by designing a screw or other mixing tools like Brabender-mixer with a gentle shear to retain fibre length. The key will be to disperse the fibres optimally while breaking as few as possible to maintain fibre length/connectivity.

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Some special techniques such a forming two phase systems and having the conductive material concentrate in the boundaries might be applied as the work continues, but in these preliminary experiments the work with be mainly empirical.

1.4.2 Process:

Preparation of filled polymers using routine techniques and compounding equipment and trained resources available at RDK

No new process technology expected to be required except that pertaining to the control of the exposure to conductive fillers, some of which e.g. conductive carbon powder, are considered potential carcinogens. Any volatile components which may escape up the vacuum vent need to be recognized and caught in a cold trap if quantities exceed allowable limits (ref. Safety Health and Enviromental Protection Manual-Section 5-20)

Some analytical techniques will be required like measurement of volume resistivity, optical or electronic microscope, mechanical properties tests.

1.4.3 Equipment

According to different experiment designs and formulations, different equipment will be used. These will be stated respectively in the individual experiment later.

1.4.4 Disposition of Samples

The best injection molded plaques will be tested and sent to Ballard.

1.4.5 Identifying and Dealing with Non Standard Conditions

The nature of this experiment means that most of the time we will be dealing with "non standard" conditions because the product and process has not yet been developed /standardized. As yet there are no standard conditions.

However, there are other "unacceptable" conditions to be aware of throughout these runs. **Some of these pertain to safety and are dealt with more fully in the Safety/Hazard Analysis section below**.

- There must be no escape of loose airborne filler particles from any part of the equipment. There is a inhalation and skin/eye irritation health hazard as well as the electrical short circuit hazard due to the fillers being highly conducting. See additional comments under Safety below.
- The high filler contents will mean difficult mold filling and high torque is a constant possibility.
- Other non standard conditions would relate to machine settings obviously not corresponding to those laid out in this operating instruction, high/low temperatures, high pressures, If these are seriously out of range, the run should be interrupted to investigate reasons and correct the problem

2. SAFETY, HEALTH PROTECTION – HAZARD ANALYSIS

ROUTINE ISSUES

1. Read the MSDS for all materials to be used
2. Read OI
3. Read Standard Practice of the equipment to be used
4. Check all ventilation ducts etc are on and creating sufficient suction to remove any expected fume

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Take precautions in handling hot polymer during compounding. Exercise care around hot surfaces – wear protective gloves. Wear necessary personal protective equipment (sleeve guards/aprons etc) and employ effective local ventilation to remove fumes/dust from the work area. If any problems cause potential dust / fume exposure wear a pre-fitted approved face mask with organic/dust filter.
In the event of absolutely needing (avoid if at all possible) to look down extruder or injection molder feed hoppers, vents, or standing in line with die/nozzle wear full face protection to guard against unexpected blow out of hot polymer, and long sleeve protection - ESPECIALLY IF DIFFICULTIES ARE BEING EXPERIENCED.
Avoid spilling resin pellets on the floor and sweep up promptly if spilled as they provide a particularly high risk for a slip / fall injury.

SPECIAL SAFETY ISSUES AND MEASURES FOR THESE EXPERIMENTS

(1) Nickel Coated carbon fibres are highly electrically conductive. Although the fibres are bundled with resin binders, there are still some short fibres or bundles which can fly in the air. Therefore, sucking vent must be installed above the fibre feeding hopper and care must be given to prevent the fibres from flying into any electrical instruments.
(2) The standard fibre size we will use in the experiment is ¼ inch long, 8-10 μM in diameter. Short NC fibres could be inhaled into human body and cause heath problems. The International Agency for Research on Cancer (IARC) concluded that metallic nickel is possibly carcinogenic to humans. Therefore, we will use the procedures outlined in OI-2442-51 (auxiliary hopper) to control possible exposure to nickel-coated fibres. The auxilliary hopper will be filled with nickel coated fibres in fume hood and installed onto the fibre feeder Engelhardt. Once compounded, the nickel-coated fibres will be ``encapsulated`` in LCP resin as such prevent direct exposure. Operators should wear mask and gloves to avoid direct skin contact with nickel coated carbon fibres. Please refer to MSDS of nickel coated carbon fibres for detailed information on safety issues.
(3) The nickel-coated carbon fibres to be used has 0.5% amino silane coupling agent and 10-20% of resin binder on the fibre surface. According to fibre supplier`s information, the minor amount of coupling agent will not cause problems during the extrusion under 300C. Care should be given to any possible thermal degradation of resin binders. If any degradation occurs, virgin PE will be used to flash the barrel.
(4) High filler loading can cause too high melt torque during the extrusion. Therefore, care should be exercised on starting. If this occurs, PE will be used to flash out the materials from the barrel.
(5) Good ventilation must be kept above fibre feeder or vent.
(6) All hazardous filler materials are to be handled in a fume hood (or other ventilated enclosure)

Related Procedures/Reference Documents:

All MSDS & Standard Practices for these equipment to be used must be on hand.

4. REGULATORY ASPECTS:

WHMIS: No controllable substances.

CEPA: report any new materials ordered: LCP, PP, Carbon fibres, Nickel-coated carbon fibres, graphite powder etc.

5. ENVIRONMENTAL IMPACT:

Minimal air / water impact via fumes extracted via the vacuum system and local ventilation system – cold trap to catch volatile components. Quantities escaping / emissions are very small - difficult to quantify. "Pump out" and "non standard" product can go to landfill as per normal polymer waste as long as the fillers are "encapsulated".

6. RELATED PROCEDURES/REFERENCE DOCUMENTS:

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MSDS, Standard Practice, Safety, Health and Environmental Manual.

7. PROCESS VARIABLES:

A detailed "run" sheet, referenced to related experimental operating instruction (O.I.) with final details and calculations must be developed from the above information and provided to the equipment operators and entered into a Laboratory Notebook.
All run details (amounts used, lot numbers, actual pressures & temperatures) as experienced, and testing results obtained are also to be entered into the Laboratory Notebook and recorded along with this O.I.
All samples are to be labeled in accordance with usual RDK practice of notebook number/page/sample

In the following formulations, Unsized Nickel-coated Carbon Fibres (UNCF) are to be used in order to get longer fibre length in compression or injection molded samples. The metal coating should help prevent from fibre breakage and improve conductivity of the composites. UNCF will be compounded with Thermocarb graphite powder (T/C) and resin (LCP) in a Brabender Plasticorder. Compression-molded samples will be made from the compounded materials.

We obtained UNCF rovings from its producer. These will be chopped to 1 inch long for feeding to Brabender.

	UNCF	T/C	LCP
2557-05-1 (40 Gram)	42% (16.8)	14% (5.6)	44% (17.6)
2557-05-2 (40 Gram)	28% (11.2)	28% (11.2)	44% (17.6)
2557-05-3 (40 Gram)	14% (5.6)	42% (16.8)	44% (17.6)
2557-05-4 (40 Gram)	51% (20.4)	17% (6.8)	32% (12.8)
2557-05-5 (40 Gram)	34% (13.6)	34% (13.6)	32% (12.8)
2557-05-6 (40 Gram)	17% (6.8)	51% (20.4)	32% (12.8)

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Brabender blending condition:
320C, speed 9, feed at first resin, then T/C and finally fibre, mix 5 min after fibre feeding

Compression molding condition:
9 gram material, 320C
Teflon film on top and bottom of mold
Preheating under 1x1000 lbs for 5 min
Regulate disc into cavity
Press under 8x1000 lbs for 10 min, then water cooling under pressure (8x1000 lbs) to 100 C, Air cooling and blowing out the water left
Open clamp and mold

Results

1. Brabender mixing

Long fibres with a few mm were visible from mixed materials. This tells us that the NC fibres can survive Brabender mixing much better than Panex or Hexcel fibres.

2. Some NC fibre bundels were still visible from the surfaces of compression molded discs
3. V-Resistivity (Table 8)

Table 8 Volume Resistivity of 2557-05-1 to 6

Sample NO.	**Volume Resistance R (Ω)**	**Volume Resistivity ρ (Ω-CM)**
	Compression-molded disc	**Factor: 2**
2557-05-1 (42%UNCF+ 14% T/C+ 44% LCP)	Thickness=1.9mm 0.03 0.09 0.08 0.05 0.12 Average=0.074	**0.15**
2557-05-2 (28%UNCF+ 28% T/C+ 44% LCP)	T=2.15mm 0.33 0.20 0.40 0.20 0.27 A=0.28	**0.56**
2557-05-3 (14%UNCF+ 42% T/C+ 44% LCP)	T=2.2mm 0.15 0.16 0.12 0.08 0.05 A=0.11	**0.22**

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2557-05-4 (51%UNCF+ 17% T/C+ 32% LCP)	T=2.3mm 0.0192 0.0318 0.0250 0.0267 0.0582 A=0.032	**0.06**
2557-05-5(1) (34%UNCF+ 34% T/C+ 32% LCP)	T=2.2mm 0.0151 0.0165 0.0192 0.0085 0.0130 A=0.0144	**0.03**
2557-05-5(2)	T=2.5mm 0.0106 0.0108 0.0040 0.0220 0.0118 A=0.0118	**0.02**
2557-05-5(3)	T=2.5mm 0.0230 0.0138 0.0096 0.0232 0.0094 A=0.016	**0.03**
2557-05-6(1) (17% UNCF+ 51% T/C + 32% LCP)	T=2.4mm 0.0146 0.0180 0.0494 0.0256 0.0230 Average=0.026	**0.05**
2557-05-6(2)	T=2.5mm 0.0152 0.0268 0.0181 0.0108 0.0065 A=0.0154	**0.03**
2557-05-6(3)	T=2.5mm 0.0280 0.0163 0.0242 0.0170 0.0127 A=0.0196	**0.04**

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Discussion

From the results listed in table 8 we can draw the following conclusions:

1. Generally speaking, higher filler loading brings better conductivity for the composites (Compare 05-1, -2, -3 with 05-4, -5, -6).
2. If the majority in the filler composition (fibre/Thermocarb) is T/C (05-3 and 05-6), the composite can still have similar conductivity as fibre has the majority (05-1 and 05-4).
3. The 05-4 with highest fibre loading shown worse conductivity than 05-5 and 05-6. This is a strange result and need to be explained later after more examination. One possible cause could be that if fibre loading is already high enough to form conductive network, adding extra T/C might disturb fibre distribution and damage network. This should be verified later.

Addendum to OI 2557-05 (I)

Because UNCF fibre are much more expensive than T/C, injection molding will be tried to use the formulation 2557-05-5 and 2557-05-6, which have shown good conductivity but with less fibre loading. These will be compounded at first in 200 gram Brabender mixer, then broken manually into small pieces retaining fibre length. In order to distinguish from compression molded samples, we give new sample code as 2557-05-5B and 2557-05-6B. In addition to that, other formulations (2557-05-07B, 2557-05-08 and 2557-05-9) are designed to use chopped and NC Prepreg in order to compare the effect of resin binder on the conductivity of the injection-molded samples.

At the same time, Stainless Steel fibres (SS) is chosen to formulate some formulations (2557-05-10B to 2557-05-15B) in order to find some good compression molded samples.

	UNCF	T/C Concentrate	LCP	Formulation
2557-05-5B	34%	52%	14%	(34%UNCF+34%T/C+
(1.5 KG)	(0.51)	(0.78)	(0.21)	32%LCP)
(7x214 Gram)	(7x72.8)	(7x111.4)	(7x30)	

(In Brabender blending for 7 batches, each batch is 214 Gram)

	UNCF	T/C Concentrate	LCP	Formulation
2557-05-6B	17%	78%	5%	(17%UNCF+51%T/C+
(1.5 KG)	(0.255)	(1.17)	(0.075)	32%LCP)
(7x214 Gram)	(7x36.4)	(7x167.1)	(7x10.7)	

(in Brabender blending for 7 batches, each batch is 214 Gram)

	NC-PP4	T/C Concentrate	LCP	Formulation
2557-05-7B	34%	52%	14%	(34%$NC+34%T/C
(1.5 KG)	(0.51)	(0.78)	(0.21)	32%LCP) PP4
(7x214 Gram)	(7x72.8)	(7x111.4)	(7x30)	

(in Brabender blending for 7 batches, each batch is 214 Gram)

	NC-PP4	T/C Concentrate	LCP	
2557-05-8	34%	52%	14%	(34%$NC+34%T/C+
(1.5 KG)	(0.51)	(0.78)	(0.21)	32%LCP) PP4

(Just dry blend for injection molding)

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	NC-PP3	T/C Concentrate	LCP	
2557-05-9 (1.5 KG)	34% (0.51)	52% (0.78)	14% (0.21)	(34%NCP+34%T/C+32%LCP)

(Just dry blend for injection molding)

The following formulations consist of Stainless Steel (SS), Thermocarb graphite powder (T/C) and resin LCP. These are to be compounded in 40 gram Brabender mixer.

	SS	T/C	LCP
2557-05-10B (40 Gram)	42% (16.8)	14% (5.6)	44% (17.6)
2557-05-11B (40 Gram)	28% (11.2)	28% (11.2)	44% (17.6)
2557-05-12B (40 Gram)	14% (5.6)	42% (16.8)	44% (17.6)
2557-05-13B (40 Gram)	51% (20.4)	17% (6.8)	32% (12.8)
2557-05-14B (40 Gram)	34% (13.6)	34% (13.6)	32% (12.8)
2557-05-15B (40 Gram)	17% (6.8)	51% (20.4)	32% (12.8)

Operation conditions and results

1. 200 gram Brabender mixing

Under the condition (set temperature=310C and speed=40rpm), 2557-05-6B was done well. The only problem was that the fibre feeding took 20 min in order to get better fibre mixing with resin and T/C. After mixing, fewer long fibres (long enough to be seen with eyes) are visible. This means the unsized chopped NC fibre was already broken during the Brabender mixing.

2557-05-5B has proved to be unsuccessful to mix, because the melted resin and T/C concentrate stuck on the mixer and didn't mix with the fibres fed later. The fibres to be fed had too larger volume than the resin.

2557-05-7B was compounded just for 10 min, because the fibre are chopped bundles, easily fed. After mixing, some unbroken fibre bundles are still visible.

2. 40 gram Brabender mixing:

320°C, Speed=scale 9;

At first fed resin, 2 min later fed T/C and then fed SS fibre slowly; mixed for 5 min after finishing feeding SS fibre; stoped motor, opened the mixer; took off the materials and broke into small pieces. After mixing, bent long SS fibres are visible. This told us the SS fibres could be bent and

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are good to survive the mixing process. Compression molded sample were made under the conditions (9 gram materials, 320C preheating for 5 mins under 1000 lbs, then press under 8000lbs for 10 min and water cooling under same pressure to 90 C). The resistivity measurement was done. The results are listed in table 9.

3. Injection molding (see attached ~~sheet 5~~ page 15 for injection molding conditions)
During the injection molding of 2557-05- 5B, the following problems arose:
(1) Very difficult feeding because of the irregular size of the compounded materials (from Brabender).
(2) Very bad flowability caused plugging of spru~~c~~e.
(3) A little smoke has been seen when using injection pressure to open the pluged spru~~c~~e. This smoke could be caused by the possible moisture from the materials or the resin binder. Requisition is sent to analytical lab for the resin binder PeOX.

Just half plaques of 2557-05-7B were obtained from this injection molding runs. 2557-05-8 and -9 were not tried. 10 gram compounded 2557-05-6B and 2557-05-7B were used to prepare compression molded samples. The resistivity of the discs are listed in table 9.

Table 9 Volume Resistivity of 2557-05-6B and 7B

Sample NO.	**Volume Resistance R (Ω)**	**Volume Resistivity ρ (Ω-CM)**
	Compression-molded disc	
2557-05-6B (1) (17%UNCF+ 51% T/C+ 32% LCP)	Thickness=3 mm 0.1725 0.2346 0.2556 0.2037 Average=0.2166	**Factor=2.5** **0.54**
2557-05-6B (2)	T=2.5mm 0.1201 0.1502 0.0952 0.3315 0.1036 A=0.1601	**Factor=2** **0.32**
2557-05-6B(3)	T=2.5mm 0.2124 0.1630 0.1058 0.0880 0.1424 A=0.1423	**Factor=2** **0.29**
2557-05-7B (1) (34[illegible]NC[illegible]+ 34T/C+ 32%LCP)	T=2.5mm 0.0019 0.0016 0.0032 0.0020 A=0.0021	**Factor=2** **0.00413**

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2557-05-7B (2)	T=2.5mm 0.0048 0.0037 0.0041 0.0047 0.0031 A=0.0041	Factor=2 0.0080
2557-05-7B (3)	T=2.5mm 0.0030 0.0053 0.0058 0.0030 0.0037 A=0.0042	Factor=2 0.008
2557-05-10B (42%~~UNCF~~ SS+ 14% T/C+ 44% LCP)	T=1.7mm 0.021 0.0185 0.0118 0.0216 0.0097 A=0.0165	Factor=1.8 0.03 (Some resin drained on compression molding)
2557-05-11B (28%~~UNCF+~~ SS 28% T/C+ 44% LCP)	T=1.9mm 0.0340 0.0609 0.0415 0.0298 0.0313 A=0.0395	Factor=1.8 0.07
2557-05-12B (14%~~UNCF~~ SS+ 42% T/C+ 44% LCP)	T=2.6mm 0.1355 0.1735 0.1700 0.1312 0.1525 A=0.1525	Factor=2 0.31
2557-05-13B (51% ~~UNCF~~ SS+ 17% T/C + 32% LCP)	T=1.8mm 0.0149 0.0180 0.0089 0.0187 0.0282 Average=0.0177	Factor=1.8 0.03
2557-05-14B (34%~~UNCF+~~ SS 34%T/C+ 32%LCP)	T=2.2mm 0.0827 0.0770 0.0690 0.0413 0.0432 A=0.0477	Factor=2 0.095

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~~Table 9 (continued)~~

(Sheet 5) as Reference for 01-2557-05—

NISSEI INJECTION MOLDING MACHINE

TIMERS

Injection	Curing	Cycle Start
14 sec.	24 sec.	1 sec.

Back Pressure	Core Temp	Cav. Temp
0 PSI	150 °C	150 °C

INJECTION VELOCITY

V3	V2	V1	VS
40 %	45 %	45 %	60 %

S2	S1	SM	SD
8 mm.	18 mm.	42 mm.	5 mm.

MOLD CLAMP

Clamp Vel.	Slow Vel.	Low Pres.	High Pres.
20 %	15 %	15 %	85 %

INJECTION PRESSURE

P3	P2	P1
62 %	62 %	62 %

TP2	S3
2 sec.	10 mm.

MOLD OPEN

Slow Vel.	Open Vel.	OT2
20 %	30 %	.1 sec.

EJECT

For. Vel.	Temp Count
10 %	.1

MOLD Family III Plaques
MAT'L. Filled LCP
01# ~~2667~~-05
DATE ~~Aug~~ Sept. 17/98
END USE Testing
CUSTOMER Yugi Cai
PROJECT# 73132

TEMPS.: MELT °F. NOZZLE 610°F. FRONT 610 °F. MID. 600°F. REAR 590 °F.

2442-65-25B : Run at above settings: light drool, min. flash. Reasonable good pick-up. Sprule sticking, release used.

2442-65-26B : Pressure up to 85', shot size 48. Sprule sticking.

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Table 9 (continued from page 16)

2557-05-15B (17% ~~UNCF~~ +SS 51%T/C+ 32%LCP)	T=2.5mm 0.0400 0.0289 0.0320 0.0590 0.0604 A=0.044	Factor=2 0.088

Discussion

In addition to resistivity measurement, optical microscopy were made to several samples in order to check the fibre length or distributions (Fig. 14 to Fig. 16) (Page 18-14)

From the results we can see

1. SS fibres can survive Brabender mixing. They were bent but not broken. The exact length is difficult to measure, because they were partly imbeded in matrix, but the longest ones are longer than 1 mm (fig 14) They should built network easier compared to carbon fibre, because carbon fibres are easy to break. Although SS fibres have at least double higher density than NC fibres, with the loading of 51% of SS (very low volume percentage loading), the resistivity is already 0.03 OHM-CM. If we raise volume percentage of SS, it will built a conductive network and bring very good conductivity. However, it is probably good just for compression molding. For injection molding it might be too heavy to distribute.
2. Comparing the conductivity of compression molded 2557-05-5 and 2557-05-7B, it is surprising that the later has 10 times better conductivity, although it has actually lower fibre loading because there is 20% resin binder in it. From optical microscopy we see many unbroken fibre bundles in 2557-05-7B (Fig 15), but not in 2557-05-5 (Fig. 16). This tells us the resin binder helped to protect fibre from breakage during Brabender mixing. These unbroken fibre bundles must have played important role in the improvement of the conductivity.
3. Fibre loading is much more important than that of T/C.
4. NC-prepreg would bring better conductivity in injection molded samples if good fibre distribution could be achieved, because the resin binder will help to prevent fibre breakage.
5. Lower filler loading should be tried in order to get better mold filling and fibre distribution.

Addendum to OI 2557-05 (II)

The above injection runs told us that the filler loading used are too high and NC-PP4 is not suitable for so high processing temperatures. NC-PP3 has 20% of resin binder (PE) on the surface. PE should be stable during the processing. Therefore, this prepreg is used for the following formulations starting with lower filler loading. From the following formulations the effect of T/C or fibre on the conductivity of the composites will be compared.

	NC-PP3	T/C Concentrate	LCP	Formulation
2557-05-16 (2000 g)	20% (400)	15% (300)	65% (1300)	(20%NCPP3+10%T/C +70%LCP)
2557-05-17 (2000 g)	30% (600)		70% (1400)	(30%NCPP3+70%LCP)
2557-05-18 (2000 g)	30% (600)	15% (300)	55% (1100)	(30%NCPP3+10%T/C +60%LCP)

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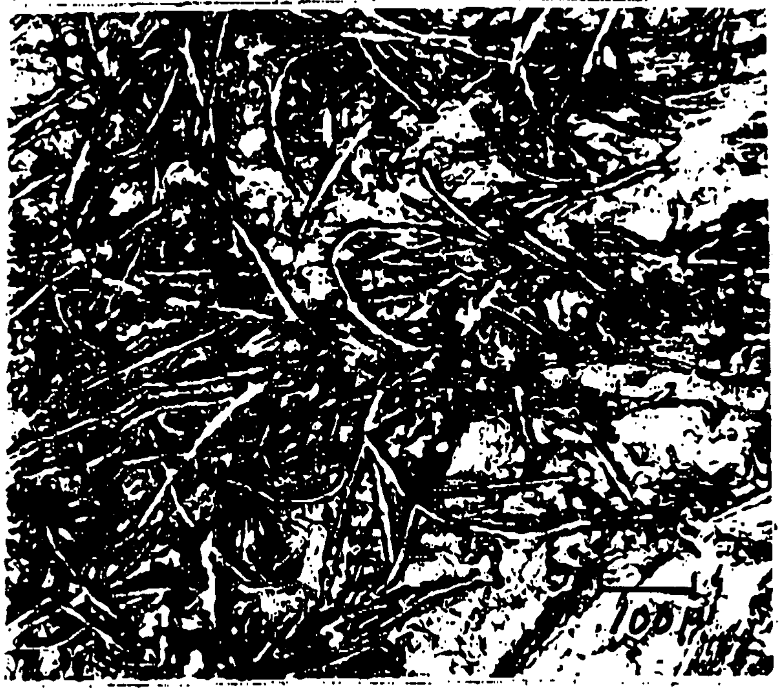

Fig. 14. Optical Microscopy of the surface of compressed disc of 2557-05-13B.

Fig. 15 Optical Microscopy of the surface of compressed disc of 2557-05-7B

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Fig. 16. Optical Micros
of the surfac
compressed
of 2557-05-

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2557-05-19 (2000 g)	40% (800)		60% (1200)	(40%NCPP3+60%LCP)
2557-05-20 (2000 g)	40% (800)	15% (300)	45% (900)	(40%NCPP3+10%T/C +50%LCP)
2557-05-21 (2000 g)	20% (400)	46% (920)	34% (680)	(20%NCPP3+30%T/C +50%LCP)
2557-05-22 (2000 g)	20% (400)	62% (1240)	18% (360)	(20%NCPP3+40%T/C +40%LCP)

Results of 2557-05-16 to 2557-05-22

1. Injection molding

In order to avoid fibre breakage, zero back pressure and possible lowest injection pressure were selected for every formulation. For injection molding conditions, please see the attached injection molding sheet (Sheet 6) (page 22). The special features occurring in the injection molding process are listed as follows:

No.	Injection pressure	Phenomena
2557-05-16	P1 20% P2 20% P3 20%	Easy to feed; good flowability; almost no spru~~c~~e plugging; some blisters in plaques
2557-05-17	16% 16% 16%	Easy to feed; good flowability; almost no spru~~c~~e plugging; some blisters in plaques
2557-05-18	16% 16% 16%	Feeding in small amount, otherwise hopper bridging; Increase material pick-up (plast. Velosi.)to 99%; Not good fibre distribution; Some blisters in plaques
2557-05-19	20% 20% 20%	Feeding in small amount, otherwise hopper bridging; Increase material pick-up (plast. Velosi.)to 99%; more blisters in plaques
2557-05-20	25% 25% 25%	Feeding in small amount, otherwise hopper bridging; Increase material pick-up (Plast. Velosi.)to 99%; Not good fibre distribution; blisters in plaques
2557-05-21	28% 28% 25%	Good feeding and mold filling; fewer blisters; Separate between fibre and T/C in plaques

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		Recorded by Caling	

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2557-05-22 28% 28% 28% same to above

2. Volume resistivity

Volume resistivity of the injection molded plaques were measured with four probe methods. See tab 10 for results.

Tab 10 V-Resistivity of 2557-05-16 to –22

Sample NO.	Volume Resistance R (Ω)		Volume Resistivity ρ (Ω-CM)	
	Injection -molded plaque (3 mm thick)		Factor=2.5	
	Parallel to flow	Vertical to flow	Parallel to flow	Vertical to flow
2557-05-16 (20%NCPP3+ 10% T/C+ 70% LCP)	>MΩ	> MΩ	>MΩ	>MΩ
25157-05-17 (1) (30% NCPP3 + 70% LCP)	0.017 0.020 0.019 0.026 0.028 A=0.022	0.011 0.019 0.020 0.025 0.014 A=0.018	0.055	0.045
2567-05-17(2)	0.014 0.032 0.026 0.027 0.036 A=0.027	0.020 0.016 0.018 0.026 0.046 A=0.025	0.067	0.063
2557-05-17(3)	0.021 0.027 0.014 0.020 0.035 A=0.023	0.011 0.026 0.021 0.013 0.019 A=0.018	0.056	0.045
2557-05-18(1) (30%NCPP3+ 10%T/C+ 60%LCP)	0.034 0.018 0.015 0.020 0.016 A=0.021	0.026 0.019 0.013 0.013 0.024 A=0.019	0.052	0.048

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Invented by Cathy

Recorded by Cathy

Date Sept 28/98

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sheet 6

NISSEI INJECTION MOLDING MACHINE

TIMERS

INJECTION	CURING	CYCLE START
14 sec.	18 sec.	1 sec.

BACK PRESSURE	CORE TEMP	CAV. TEMP.
~0 psi	150 oC	150 oC

INJECTION VELOCITY

V3	V2	V1	V5
25 %	30 %	30 %	50 %

S2	S1	S1	SD
8 mm.	18 mm.	46 mm.	5 mm.

MOLD CLAMP

CLAMP VEL	SLOW VEL	LOW PRES
20 %	15 %	15 %

HIGH PRES
85 %

INJECTION PRESSURE

P3	P2	P1
16 %	16 %	16 %

TP2	S3
2 sec.	10 sec.

MOLD OPEN

SLOW VEL	OPEN VEL	OT2
20	30	.1

MOLD Spinney II
MAT'L Heavily Filled LCP
OI # 2557-05
DATE Sept 28/98
END USE Testing
CUSTOMER Yuqi Cai
PROJECT # 73132

EJECT

FOR. VEL	TEMP. COUNT
10 %	1

TEMPS:

MELT 606 oF FRONT 600 oF REAR 590 oF
NOZZLE 600 oF MID 600 oF

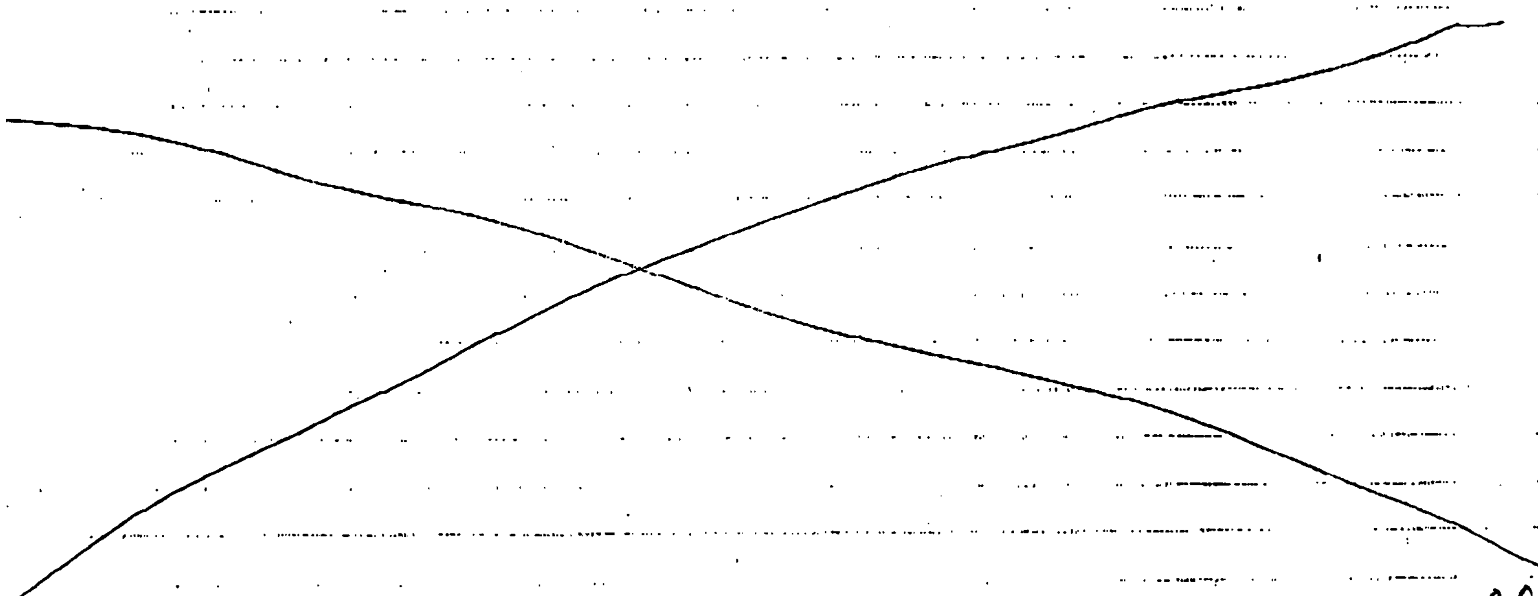

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Table 10
(Continued from page 21)

2557-05-18(6)	0.011 0.017 0.014 0.008 0.014 A=0.013	0.015 0.009 0.017 0.013 0.012 A=0.013	0.033	0.033
2557-05-18(8)	0.026 0.020 0.020 0.021 0.012 A=0.020	0.015 0.019 0.024 0.010 0.015 A=0.017	0.050	0.041
2557-05-19(1) (40%NCPP3+ 60%LCP)	0.008 0.016 0.018 0.010 0.010 A=0.012	0.009 0.015 0.011 0.009 0.005 A=0.01	0.030	0.025
2557-05-19(3)	0.008 0.008 0.014 0.016 0.01 A=0.011	0.007 0.008 0.014 0.007 0.01 A=0.009	0.028	0.023
2557-05-19(5)	0.007 0.009 0.009 0.012 0.013 A=0.01	0.015 0.009 0.004 0.009 0.009 A=0.009	0.025	0.023
2557-05-20 (1) 40%NCPP3+ 10%T/C+ 50%LCP)	0.021 0.009 0.019 0.014 0.016 A=0.016	0.011 0.012 0.012 0.021 0.009 A=0.013	0.039	0.033
2557-05-20 (5)	0.012 0.007 0.010 0.010 0.007 A=0.011	0.008 0.012 0.01 0.006 0.008 A=0.009	0.028	0.022
2557-05-20 (6)	0.014 0.013 0.016 0.017 0.006 A=0.013	0.009 0.007 0.011 0.009 0.011 A=0.094	0.033	0.023

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2557-05-21 (4) (20%NCPP3+ 30%T/C+ 50%LCP)	0.040 0.044 0.072 0.024 0.039 A=0.04	0.029 0.050 0.054 0.033 0.019 A=0.037	**0.10**	**0.09**
2557-05-21 (7)	0.020 0.016 0.019 0.021 0.016 A=0.016	0.017 0.017 0.034 0.011 0.030 A=0.021	**0.04**	**0.05**
2557-05-21 (9)	0.028 0.026 0.025 0.021 0.018 A=0.024	0.026 0.020 0.018 0.031 0.021 A=0.023	**0.059**	**0.058**
2557-05-22 (1) (20%NCPP3+ 40%T/C+ 40%LCP)	0.031 0.018 0.026 0.022 0.035 A=0.026	0.026 0.045 0.033 0.019 0.029 A=0.030	**0.066**	**0.075**
2557-05-22 (4)	0.022 0.013 0.023 0.040 0.021 A=0.024	0.028 0.034 0.019 0.018 0.025 A=0.025	**0.060**	**0.065**

Discussion

1e results of volume resistivity showed that with 40% NCPP3 we can achieve the resitivity of 02-0.03 which is very close to our target 0.01.

crease of fibre loading from 30% to 40% caused revolutionary change in resistivity (comparing 7 and 19). This tells us the percolation point is in the range of 30 to 40%.

'C powder does help build conductive network if the fibre loading alone is not high enough to rm the network. This is verified in comparing 16, 21 and 22.

the fibre loading is already high enough to form a network, adding T/C powder to the fibres besn't help bring better conductivity. Even it will affect the conductivity because it will disturb ore's flow and distribution.

listers in plaques told us that lower processing temperatures should be used or other resin nders should be chosen because the blisters were probably from the resin binder PP3.

Addendum to OI 2557-05 (III)

he following formulations will be tried under lower injection molding temperatures in order to heck the cause of blisters in the samples done last time. The materials are dried at 102C for 38

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hrs. At the same time the NC-prepreg with other resin binders will be tried.

	NC-PP3	T/C Concentrate	LCP	Formulation
2557-05-19R (2000 g)	40% (800)		60% (1200)	(40%NCPP3+60%LCP)

	NC-PP6	T/C Concentrate	LCP	Formulation
2557-05-23 (2000 g)	40% (800)		60% (1200)	(40%NCPP6+60%LCP)

	NC-PP1	T/C Concentrate	LCP	Formulation
2557-05-24 (2000 g)	40% (800)		60% (1200)	(40%NCPP1+60%LCP)

Results

<u>Injection molding (see attached sheet 7 (page 27) for injection molding conditions)</u>

1. Feeding was good only if small amount of materials were fed every time., otherwise the fibres would form bridging and stop the feeding.
2. The same injection pressure was used for all the three formulations. Mold filling had no problem.
3. Although lower barrel temperatures (than last time) were used, there were still many blisters or voids in plaques of 2557-05-19R and 2557-05-24. In plaques of 2557-05-23 there were fewer blisters. The sample surfaces looked much better.

<u>Resistivity of the injection molded plaques</u>

Volume resistivity of some plaques were measured with four probe methods. See table 11 for results.

Tab 11 V-Resistivity of 2557-05-16 to –22

Sample NO.	**Volume Resistance R (Ω)**		**Volume Resistivity ρ (Ω-CM)**	
	Injection –molded plaque (3 mm thick)		Factor=2.5	
	Parallel to flow	Vertical to flow	Parallel to flow	Vertical to flow
2557-05-19R(2) (40%NCPP3+ 60% LCP)	0.0187 0.0350 0.0120 0.0260 0.0350 A=0.025	0.015 0.018 0.009 0.014 0.012 A=0.014	**0.063**	**0.034**

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		Recorded by [illegible]	

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2557-05-19R(3)	0.0129 0.0145 0.0143 0.0182 0.0270 A=0.0174	0.0140 0.0130 0.0150 0.0390 0.0104 A=0.0182	**0.043**	**0.046**
2557-05-23(3) (40%~~C~~NPP6+ 60%LCP) NCPP6	0.0136 >MΩ >MΩ >MΩ A=?	0.0145 0.0034 0.0052 0.084 >MΩ A= ?	?	?
2557-05-23(4)	>MΩ 0.0090 0.0152 0.0139 >MΩ A=?	>MΩ 0.0077 >MΩ 0.0036 0.0072 A=?	?	?
2557-05-24(1) (40%NCPP1+ 60%LCP)	Reading changed from MΩ→0.003	Reading changed from MΩ→0.003	?	?
2557-05-24(2)	Reading changed from MΩ→0.003	Reading changed from MΩ→0.003	?	?

Discussion

1. Although lower barrel temperature were chosen and the materials were dried before injection molding, there were still many blisters in 19R and 24. This means the temperature is still too high for these two sorts of resin binders (PP3 and PP1). There were much fewer blisters in 23. This tells us that the resin binder PP6 has much higher thermal stability.
2. 19R gave a little worse conductivity than last time (19). This means processing conditions have influence. Lower processing temperature might have affected the fibre distribution. This is to be verified using microscope.
3. 23 and 24 showed strange phenomina above on measuring the resistance. These could result from that the fibre loading is very close to the percolation point. In order to verify that, we need increase fibre loading to see how the conductivity changes.

Suggestion
[redacted] to OI 2557-05 ([redacted])

1. Lower processing temperatures will be used if we use the same LCP or we chose other LCP resin with lower melting point.
2. Higher fibre loading will be used in the systems like 23 and 24.

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<< sheet 7 >>

NISSEI INJECTION MOLDING MACHINE

TIMERS

INJECTION	CURING	CYCLE START
14 sec.	18 sec.	1 sec.

BACK PRESSURE	CORE TEMP.	CAV. TEMP.
-0- psi	150 oC	150 oC

INJECTION VELOCITY

V3	V2	V1	V5
18 %	18 %	18 %	65 %

S2	S1	S1	S0
8 mm.	18 mm.	56 mm.	5 mm.

MOLD CLAMP

CLAMP VEL.	SLOW VEL.	LOW PRES.
20 %	15 %	15 %

HIGH PRES
95 %

44 Plaques
8 Tensiles

INJECTION PRESSURE

P3	P2	P1
20 %	20 %	20 %

TP2	S3
2 sec.	10 sec.

MOLD Family III
MAT'L Heavy Filled LCP
OI # 2557-05
DATE Oct. 1/98
END USE Testing
CUSTOMER Vuei Cal
PROJECT # 73132

MOLD OPEN

SLOW VEL	OPEN VEL	OT2
20	30	.1

EJECT

FOR. VEL	TEMP COUNT
10 %	1

TEMPS:

MELT 602 oF FRONT 590 oF REAR 580 oF
NOZZLE 590 oF MID 590 oF

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		Recorded by [signature]	

Book No. 2557 TITLE

No. 26

Issue date: Oct 14. 1998 by Y. Cai

OI 2557-28 Compounding of NCPP3/LCP on W&E twin screw extruder

This OI is addendum to OI 2557-05. The general information and applicable features from OI 2557-05 are still valid for OI 2557-28. Only the differences and new issues will be highlighted as following.

1.PURPOSE

W&E extruder is chosen to compound NCPP3 filled LCP, because W&E extruder can make much more gentler mixing than W&P twin screw extruder. A single strand die will be used on W&E. The extrudate will be cooled in air and pelletized for injection molding. 2 KG pellets will be prepared for each formulation.

2. SPECIAL SAFETY ISSUES AND MEASURES FOR THESE EXPERIMENTS

(1) Nickel Coated carbon fibres are highly electrically conductive. Although the fibres are bundled with resin binders, there are still some short fibres or bundles which can fly in the air. Therefore, sucking vent must be installed above the fibre feeding hopper and care must be given to prevent the fibres from flying into any electrical instruments.

(2) The standard fibre size we will use in the experiment is ¼ inch long, 8 μM in diameter. Short NC fibres could be inhaled into human body and cause heath problems. The International Agency for Research on Cancer (IARC) concluded that metallic nickel is possibly carcinogenic to humans. Therefore, we will use the procedures outlined in OI-2442-51 (auxiliary hopper) to control possible exposure to nickel-coated fibres. The auxilliary hopper will be filled with nickel coated fibres in fume hood and installed onto the fibre feeder Engelhardt. Once compounded, the nickel-coated fibres will be ``encapsulated in LCP resin as such prevent direct exposure. Operators should wear mask and gloves to avoid direct skin contact with nickel coated carbon fibres. Please refer to MSDS of nickel coated carbon fibres for detailed information on safety issues.

(3) The nickel coated carbon fibres NCPP3 to be used has 0.5% amino silane coupling agent and 20% polyethylene resin binder on the fibre surface. According to fibre supplier`s information, the minor amount of coupling agent and polyethylene resin binder will not cause problems during the extrusion under 300C. Care should be given to any possible thermal degradation of these materials. If any degradation occurs, virgin PE will be used to flash the barrel.

(4) High filler loading can cause too high melt torque during the extrusion. Therefore, care should be given on starting. If this occurs, PE will be used to flash out the materials from the barrel.

(5) Good ventilation must be kept above the resin hopper and fibre feeder or vent.

(6) All filler materials are to be handled in a fume hood (or other ventilated enclosure)

3. PROCESS VARIABLES

(1) Formulations

OI-No.	NCPP3	LCP
2557-28-1	40%	60%
2557-28-2	50%	50%

If the filler loadings are too high and cause die plugging problem, please try the following formulations:

	NCPP3	LCP

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A. Harbourne	19Oct98	Recorded by Caiy.	

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2557-28-3	20%	80%
2557-28-4	30%	70%

(2) Operation Conditions

Feeding
Engelhardt Vibra (FB) will be used for feeding resin quantitatively into resin hopper. Engelhardt Vibra & Belt (FA) with a auxiliary hopper will be used to feed fibres into the front vent hopper.

Screw configuration: Special ``D``
Die type: Single hole (Med.)
Temperatures:

Zone 1 (Rear)	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6
°F 440	540	555	555	555	555
	Zone S1	Zone S2	Zone S3	Zone S4	Die
	555	555	540	540	540

Extrudate cooling
If the extrudate can not be well conveyed just after air cooling, please use water cooling.

Procedures (refer to Standard Practice)

For 2557-28-1
-Clean the machine using PE
-Virgin LCP to flash the barrel
-Feed LCP resin to reach the output of 6 LBS/hr
-Regulate the fibre feeding rate to reach the total output (LCP+Fibre) of 10 LBS/hr

For 2557-28-2
-Clean the machine using PE
-Virgin LCP to flash the barrel
-Feed LCP resin to reach the output of 5 LBS/hr
-Regulate the fibre feeding rate to reach the total output (LCP+Fibre) of 10 LBS/hr

If the filler loading s are too high and cause die plugging problem, try 2557-05-3 and 2557-05-4.
For 2557-28-3
-Clean the machine using PE
-Virgin LCP to flash the barrel
-Feed LCP resin to reach the output of 6 LBS/hr
-Regulate the fibre feeding rate to reach the total output (LCP+Fibre) of 7.5 LBS/hr

For 2557-28-4

-Clean the machine using PE
-Virgin LCP to flash the barrel
-Feed LCP resin to reach the output of 6 LBS/hr

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...ate the fibre feeding rate to reach the total output (LCP+Fibre) of 8.6 LBS/hr

...hat, cleaning the barrel using PE and the fibre hopper using special vacuum.

Results

- See run sheet (Sheet 8) for extrusion conditions
- 2557-28-1 and 2557-28-2 were tried but proved to be unsuccessful because of die plugging due to too high fibre loading. 2557-28-3 and 2557-28-4 were well done, although the later had sometimes surge on extrusion. No water cooling was used. The extrudates were conveyed manually.

W & E Extruder 20 mm

sheet 8

Date: OCT. 14/98

Operator: E. NIELSEN

Die Type: SINGLE HOLE (MED.)

Screw Configuration: SPECIAL "D" UNMATCHED

Experiment #: 2557-28 Project #: 73132

Resin Info: LCP 8000, BATCH 3011, LOT 6-4-97

	Time:	11:00		13:00								
Sample#		3		4								
		Set Pt	Actual	Set Pt	Actual	Set Pt	Actual	Set Pt	Actual	Set Pt	Actual	Set Pt	Actual
(° F)	Zone 1	440	440		435								
	Zone 2	540	543		442								
	Zone 3	555	551		550								
	Zone 4	555	552		550								
	Zone 5	555	552		552								
	Zone 6	555	555		545								
	Zone S1	555	556		554								
	Zone S2	555	554		552								
	Zone S3	540	542		543								
	Zone S4	540	535		539								
Die Setting (° C)		280	280		280								

Melt Temp:						
Screw rpm:	290	290				
Motor amps:	2.3	2.3				
Discharge psi:	500	500				
Vacuum psi:	OFF	OFF				
Throughput:						
LCP Feeder 1	6 lb/hr	6 lb/hr				
FIBRE Feeder 2	1.5 lb/hr	2.58 lb/hr				
% FIBRE	20%	30%				
Total:	7.5 lb/hr	8.58 lb/hr				

Comments: INITIALLY TRIED TO RUN @ 40% FIBRE. DIE APPEARED TO BE PLUGGING. REDUCED FIBRE TO 20%. FOR 2ND RUN INCREASED FIBRE TO 30%
2557-28-3
2557-28-4

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